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<p>Intelligent Processing of Materials concepts that combine process modeling and simulation, <i>in-situ</i> sensors, and closed-loop control were adapted and demonstrated on a vertical Bridgman (VB) for infrared (IR) and electro-optic materials. Models to simulate VB growth were developed. These models were used to investigate the dependence of the solid-liquid interface shape and thermo-mechanical stresses in the crystal on furnace temperature profile, ampoule geometry and support, and growth rate. Two types of sensors were investigated: an IR sensor and an eddy current sensor. The experimental results showed that it is not possible to image the solidification front during growth with an IR sensor through the In: CdTe crystal because of the low optical transmission and high thermal emission of In: CdTe at high temperatures. Changes in</p> <p style="text-align: center;">(continued on reverse side)</p>			
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electrical conductivity become measurable using an encircling type eddy-current sensor when the processing temperature exceeds 600°C. The eddy current sensor response can be used to monitor specimen quality. As a result, the eddy current sensor is critical for development of a flexible growth system to produce the material under favorable growth conditions and ensure reliable delivery with low cost. The potential applications of the technologies include infrared focal plane arrays and electro-optic modulators for CO₂ and surgical lasers.

**MODEL-BASED PRODUCTION OF INFRARED AND OPTICAL
MATERIALS**

FINAL REPORT

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1.0 PROGRAM OBJECTIVES

The objectives of this program were to develop and demonstrate model-based, advanced vertical Bridgman (VB) processing for infrared (IR) and electro-optic materials, employing Intelligent Processing of Materials (IPM) concepts that combine process modeling and simulation, *in-situ* sensors, and adaptive control to achieve significant improvement in crystal reproducibility and product performance. This included:

- Development and adaptation of conduction, convection and thermal stress models for VB growth.
- Numerical investigation of the dependence of the solid-liquid interface shape and thermo-mechanical stresses in the crystal on furnace temperature profile, ampoule geometry and support, crystal size and growth rate.
- Design and execution of growth experiments in a production furnace at II-VI, Inc. for model validation.
- Experimental investigation of the optical properties and imaging of In:CdTe at elevated temperatures with IR sensors, and investigation of the electrical conductivity at elevated temperatures with eddy-current sensors.

2.0 KEY RESULTS OF THE PROGRAM

The significant research findings during this program period are described below.

- In general, the growth interface is initially convex and starts turning concave beyond one-half crystal radius. The interface shape becomes fully concave beyond one crystal radius, and the degree of concavity decreases as the furnace temperature gradient increases.
- High thermal stresses are concentrated near ampoule wall and crystal center along growth interface, near ampoule support, and in the vicinity of transition from gradient zone to cold zone.

- As the hot zone temperature decreases towards the melting temperature, the interface shape becomes less concave and the stress level decreases. As the cold zone temperature increases, the interface shape becomes more concave and the stress level increases.
- The advantage of a spherical ampoule base over a flat one is a marked reduction in stress level not only during early stage of growth but also throughout the entire crystal.
- A significant reduction in thermal stress level occurs when the ampoule base is in direct contact with a solid block of material. The higher the thermal conductivity of the insert material, the lower the stress level.
- The larger the crystal diameter, the more severe the thermal stress level, in particular, at locations along the growth interface near the ampoule wall and in the vicinity of the transition point from the gradient zone to the cold zone.
- The higher the growth rate, the more concave the interface shape and, consequently, the more severe the thermal stress level.
- Convection in the melt has a typical velocity on the order of 1 mm/sec and exhibits a flow pattern of two major counter-rotating cells, one near the free surface and the other near the interface. The flow is stronger during early stage of growth because the pool is deeper, and the interface tends to be more convex because the flow cells are closer to the ampoule wall.
- Growth experiments indicate that a large amount of supercooling, as much as 30°C, is present in the melt along the interface. The results have also validated the models developed for parametric studies involving ampoule base insert materials with different thermal conductivities.
- It is not possible to image the solidification front during growth with an IR sensor through the In:CdTe crystal because of the low optical transmission and high thermal emission of In:CdTe at high temperatures.

- Changes in electrical conductivity become measurable using an encircling type eddy-current sensor when the processing temperature exceeds 600°C. Electrical conductivity remains constant at a holding temperature above 600°C.

- Furthermore, the electrical conductivity of a specimen varies with the sensor sweep frequency during cool down, indicating that a correlation exists between the sensor signals and the electrical and optical properties of the specimen.

- The eddy-current sensor response can differentiate one specimen of good quality from one of bad quality; therefore, the sensor may be useful for rapid sorting of specimens.

- The annealing and quenching process cannot change the inherent defects in an In:CdTe crystal during the growth process, but can lower the carrier concentration and increase the electrical resistivity.

- It was demonstrated that the eddy current sensor can be utilized to provide a screening method for qualification of the optical properties of In:CdTe, and can be installed in-situ for the annealing process.

These findings have enhanced fundamental understanding of the process and have established the basis for more effective process windows to produce single crystals under favorable growth conditions and, therefore, to improve crystal yield and product performance. These findings also permit the development and design of a more flexible growth system for optimum controllability. As a result, delivery of materials with high reproducibility and low cost can be realized. The potential applications of the technologies include infrared focal plane arrays and electro-optic modulators for CO₂ and surgical lasers.

3.0 PUBLICATIONS AND TECHNICAL REPORTS

None.

4.0 PARTICIPATING SCIENTIFIC PERSONNEL

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5.0 REPORT OF INVENTIONS

None.